N63-18397

OTS PRICE

XEROX $

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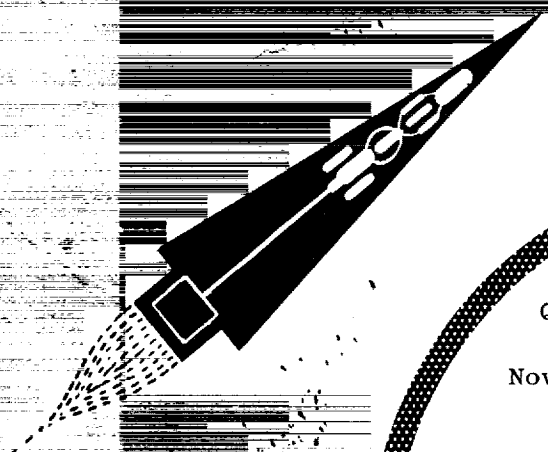


QUARTERLY REPORT NUMBER ONE

Nov 25, 1961 to Feb. 25, 1962

DM62-94

EVALUATION OF A HIGH STRENGTH COLUMBIUM ALLOY (AS-55) FOR ALKALI METAL CONTAINMENT

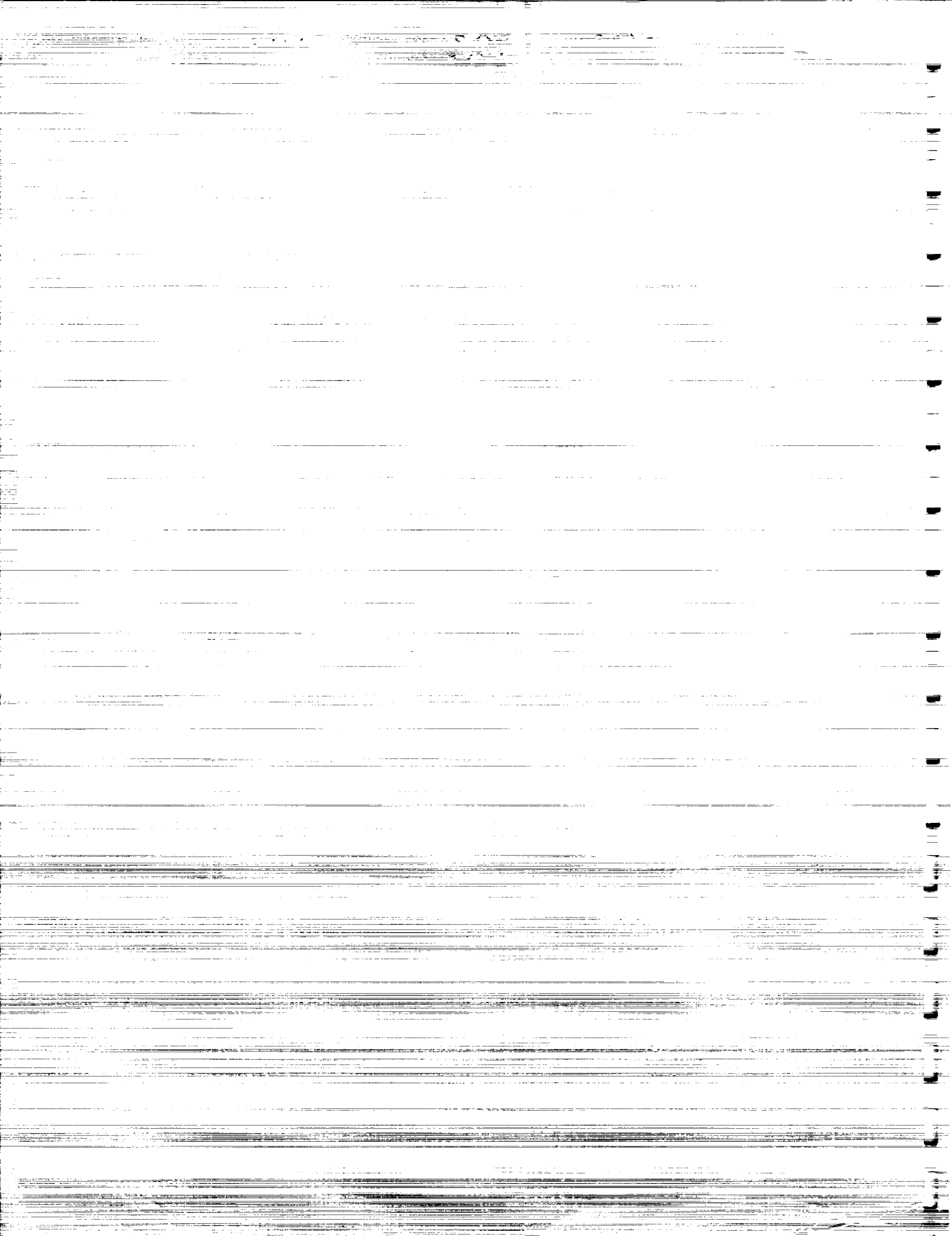
Under Contract NAS 3-2140

For

THE NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

FLIGHT PROPULSION LABORATORY DEPARTMENT

GENERAL ELECTRIC



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SPACE POWER OPERATION
FLIGHT PROPULSION LABORATORY DEPARTMENT
GENERAL ELECTRIC COMPANY
CINCINNATI 15, OHIO

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EVALUATION OF A HIGH STRENGTH COLUMBIUM ALLOY (AS-55) FOR ALKALI METAL CONTAINMENT

I. INTRODUCTION

This investigation was undertaken to obtain additional documentation of the AS-55 columbium base alloy as an alkali metal containment material for nuclear turboelectric space power systems. The AS-55 designation represents columbium base alloys with 5 to 10% W, 0.8 to 1.2% Zr, and 0.04 to 0.08% C which are arc melted with additions of 0.2 to 1% Y to the electrode, much of the yttrium being removed during melting. Based on initial tests, the AS-55 alloy possesses good elevated temperature strength, is readily fabricated and welded, and is resistant to attack by potassium.

The objectives of the program are (1) to investigate the arc melting characteristics of the alloy in order to provide material of high quality for evaluation and to prepare for the possibility of future scale-up efforts, (2) to obtain additional data on the fabricability, weldability, and mechanical behavior, particularly the long time stress-rupture properties, and (3) to investigate the corrosion resistance to potassium under refluxing conditions.

II. SUMMARY

Detailed technical plans for the program have been completed, and an outline of each portion of the work is contained in the body of the report.

Procurement of materials to be used in the program, for all practical purposes, has been completed. This includes the raw materials and mill products for electrode preparation, melting, conversion of ingot to sheet, and evaluation of the final product. The bulk of the columbium was procured as -35 mesh powder from Kennametal, Inc. and contains 0.06% C, 0.06% O, and 0.03% N. A small quantity of columbium spheres produced by the Du Pont Company, containing 0.003% C, 0.0015% O, and 0.0003% N, was also obtained. Cb-1Zr alloy sheet to be used for a comparison of the corrosion behavior has been received.

All the electrode sections required for the program were cold pressed and vacuum sintered at 2800°F. The blended alloy mixes containing the columbium powder were hydrostatically pressed into approximately 1-3/4" diameter x 10" sections, and the blend containing the columbium spheres was mechanically pressed into a $1\frac{1}{2}$" x $1\frac{1}{2}$" cross section. Of the fifteen ingots to be melted from these electrodes, nine have been completed.

Four ingots were sectioned longitudinally for evaluation and preliminary conversion trials. Billets from two of the ingots were successfully forged, warm rolled, and subsequently cold rolled to 0.060" thick sheet. Billets from the other two ingots are being processed.

An ultra-high vacuum chamber, to be used for conducting the corrosion tests, has been vacuum checked in the 10^{-10} torr range, and a capsule heater which was constructed to a preliminary design is being evaluated.

III. TECHNICAL PLANS

The technical plans which were established during the first month of the program are presented below.

A. Arc Melting Investigation

Designed requirements, requiring 14 ingots will be conducted to study the main effects of melting variables and their interactions on ingot quality. The variables to be controlled are arc voltage, arc current, stirring field, melting atmosphere (He-A mixture), and electrode-to-mold area ratio. The effects of varying the melting conditions will be evaluated by examining the ingot surface, porosity, chemistry, segregation, macro and microstructures, and hardness. Twelve ingots will be used for this portion of the investigation. In order to determine the effect of yttrium on melting, two additional ingots will be melted, i.e., one ingot will be made using the Du Pont columbium of very low interstitial element content, and the other ingot will be made using the Kennametal columbium with a lower percentage of yttrium.

1. Materials - The raw materials to be used in this work are described in Table I.
2. Electrode Preparation - The electrodes, with the exception of the one to be produced from the low interstitial element columbium, will be made by blending and hydrostatically pressing the powders to produce bars, 1-3/4" diameter x 10" long, weighing approximately 6 pounds. The pressed electrodes will be wrapped in tantalum foil and vacuum sintered at 2800°F for about one hour. A total of twenty-one 6-pound electrode sections will be produced by this method, with an anticipated density of 80% of that theoretically obtainable.

Table I

RAW MATERIALS

Element	Source	Form	Analysis
Columbium	Kennametal (Lot Bl. 287)	-35 Mesh	Cb, 99.7; C, 0.06; 0, 0.06; N, 0.03
Columbium	DuPont (Lot D-3-214)	Spheres	Cb, 99.85; C, 0.003; 0, 0.0015; N, 0.0003
Tungsten	General Electric (Lot U-18.5-3356)	18.5 Micron	W, 99.9 min.
Zirconium Hydride	Metal Hydrides (Grade R Lot S-28)	-400 Mesh	Zr, 97; H , 2 1; 0, 0.14; C, 0.02; N, 0.0037
Yttrium Hydride	FPLD*	-20 Mesh	Y, bal.; H, 3.14; 0, 1.92; N, 0.07

Table II

RANGE OF MELTING VARIABLES

Variable	Low	Midpoint	High
Voltage (volts)	X-4	X	X + 4
Current (amperes)	Y-500	Y	Y + 500
Stirring (oersteds)	Z-15	Z	Z + 15
Electrode/Mold Area Ratio	0.23	0.34	0.49

Note: The midpoint values subsequently established are:

X = 28 volts
Y = 5750 amperes
Z = 20 oersteds

The electrode with high purity columbium will be produced by mechanically die pressing the Du Pont Company columbium spheres with the alloying additions into 1-1/2" x 1-1/2" x 25" bars and vacuum sintered at 2800°F for about one hour.

Electrode sections will be joined together in the arc melting furnace by a butt sintering technique previously developed during melting studies of the F-48 columbium alloy.

3. Melting Study - After establishing approximate midpoints for melting by vacuum consumable arc melting one 12-pound ingot in a 3" diameter mold, the levels of the variables for the balance of the study will then be fixed. The range of levels selected for study are shown in Table II.

In addition, because of the effects of the low electron work function of yttrium on the melting power and arc stability in a vacuum, one electrode will be melted in an atmosphere of A+He for comparison. Another electrode with a lower yttrium content (0.2% versus 1.0%) will be melted in a vacuum.

The degree of reaction of yttrium with the interstitial elements during melting, particularly with oxygen, will be studied further by melting the nominal AS-55 composition in a vacuum, but using the high purity columbium.

The outline of the melting program is shown in Figure 1. All ingots will be hot topped by slowly reducing the stirring to zero and the current to 1,500 amperes. The hot top procedure established on the first ingot will be used on all subsequent ingots.

Figure 1

AS-55 INGOT MELTING PLAN

Variable	Range of Variables Low	Midpoint	High
(To Determine Approx. Midpoint)		(2) *	
Voltage (X)	X-4	(2) *	X+4
Current (Y)	Y-500		Y+500
Stirring (Z)	Z-15		Z+15
Electrode/Mold Area Ratio = E/M	E/M= 0.23	(2)	E/M= 0.49
Partial Pressure Inert Gas (A+He)			50 mm Hg
DuPont High Purity Cb		(2) *	
Y Variation (0.2%)		(2) *	
Optimum Melt		X_1, Z_1, Y_1, E/M = 0.34 *	

Note: (1) All melts will be made under the best vacuum attainable except the one melt under static pressures of inert gas.

(2) All variables except where noted will be midpoint conditions:

X volts
Y amperes
Z oersteds

E/M = 0.34
Kennametal Cb+1%Y

(3) *Denotes 12-pound heat.

4. Evaluation of Ingots - All ingots will be inspected for surface imperfections. Subsequently, the ingots will be cut lengthwise, and the exposed surface of one half will be ground and macro-etched. By macro and microscopic examination of this surface, a measure of porosity, grain size, and grain configuration will be obtained. Hardness measurements will also be made. Samples for chemical analyses will be taken from the top, center, and bottom sections of processed ingots after initial converstion to plate.

B. Conversion of Ingot to Sheet

Established conversion techniques, e.g., rolling and forging, will be employed. Protection against oxygen contamination during the initial high temperature breakdown of the ingot will be provided by jacketing the billets in titanium and a titanium alloy. Billets, about 1" x 2" x L , will be machined from one half of the longitudinally sectioned ingots and conditioned. Subsequently, the conditioned billets will be framed in 1" x 1" titanium bars, and cover plates of 6Al-4V-Ti sheet will be welded to the frame. The resultant pack will be evacuated and sealed in the electron beam welding facility.

Initial conversion will be accomplished by forging and/or rolling at a temperature in the range of 2200°F for the first 50 to 80% reduction of the AS-55 core thickness. The titanium frame will then be cut from the AS-55 plate, and the cladding will be stripped by a pickling operation. The exposed surface of the AS-55 material will be conditioned for further rolling, and test sections will be removed for chemical analyses, microstructure, and hardness investigations. The need for an intermediate anneal will be established.

After conditioning, additonal work will be imparted to the AS-55 plate by cold rolling to 0.060" thick sheet, approximately 90% cold reduction. A proper final anneal will be selected after examination of the microstructure and hardness of as-rolled samples and samples given various thermal treatments. The conversion flow sheet is shown in Figure 2. Conversion of additional billets from succeeding ingots will be based on the foregoing process with modifications inserted at appropriate intervals if it is considered advisable to take better advantage of the inherent good fabricability of the material. These modifications may include a less elaborate high temperature protective technique and a lower breakdown temperature, including a direct rolling operation. The most promising techniques will be used to produce a quantity of sheet material for property evaluation.

C. Evaluation of AS-55 Sheet

1. Mechanical Properties - Selected material from the conversion investigation will be evaluated in tension at room temperature and 2200°F. Other tensile data will be obtained from one heat at 1350°, 1500°, 1800°, 2000°, 2200°, and 2500°F in a vacuum.

   Vacuum stress-rupture tests are planned in order to establish 10, 100, and 1,000 hour strengths at temperatures of 1800° and 2200°F. Additional stress-rupture testing will be conducted at 2500°F for times up to 1,000 hours, depending upon the limitations of the testing equipment.

   Concurrent microstructural and hardness studies will be carried out on sheet samples from all phases of the mechanical property evaluation, (Figure 3).

Figure 2

INGOT CONVERSION FLOW SHEET

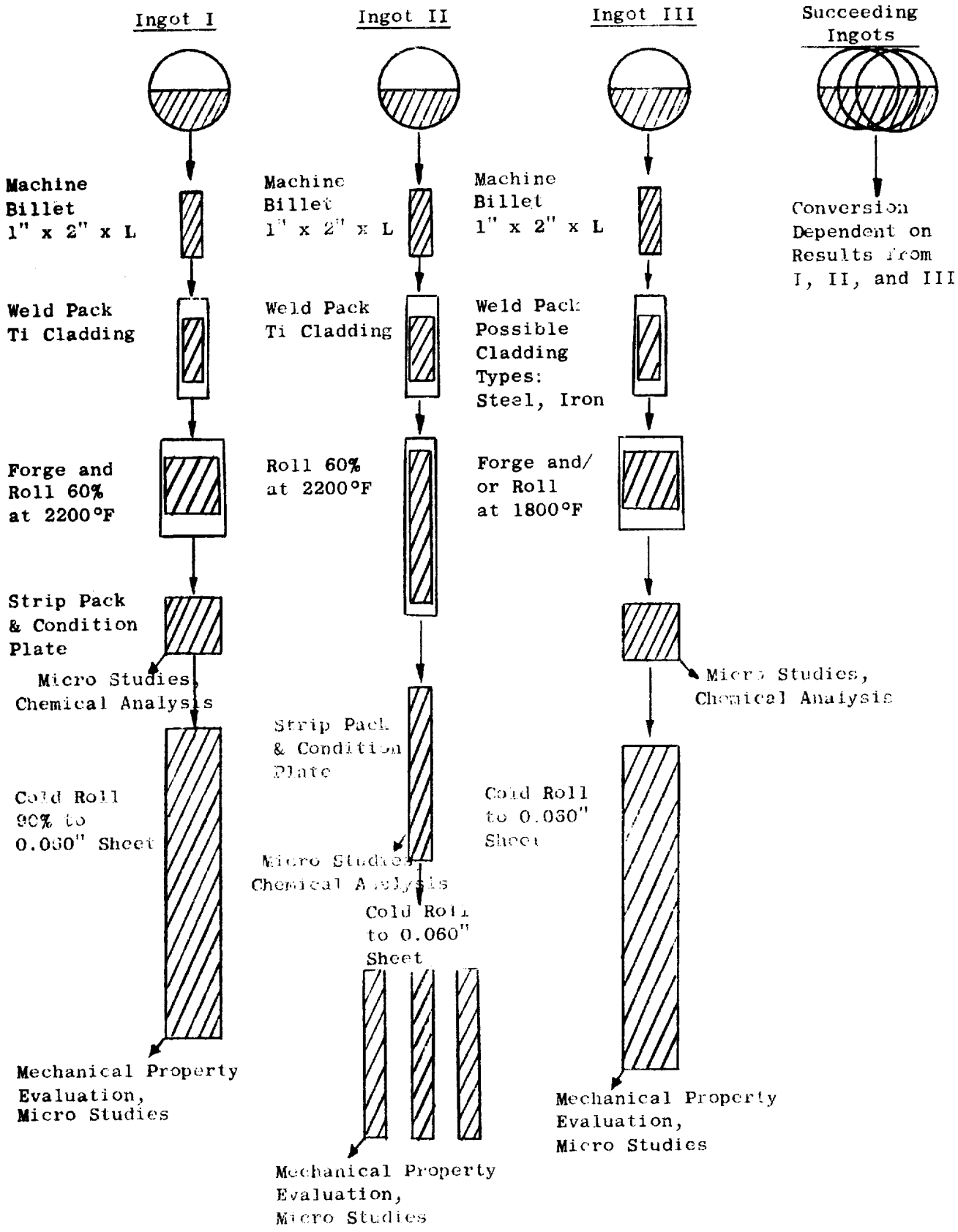
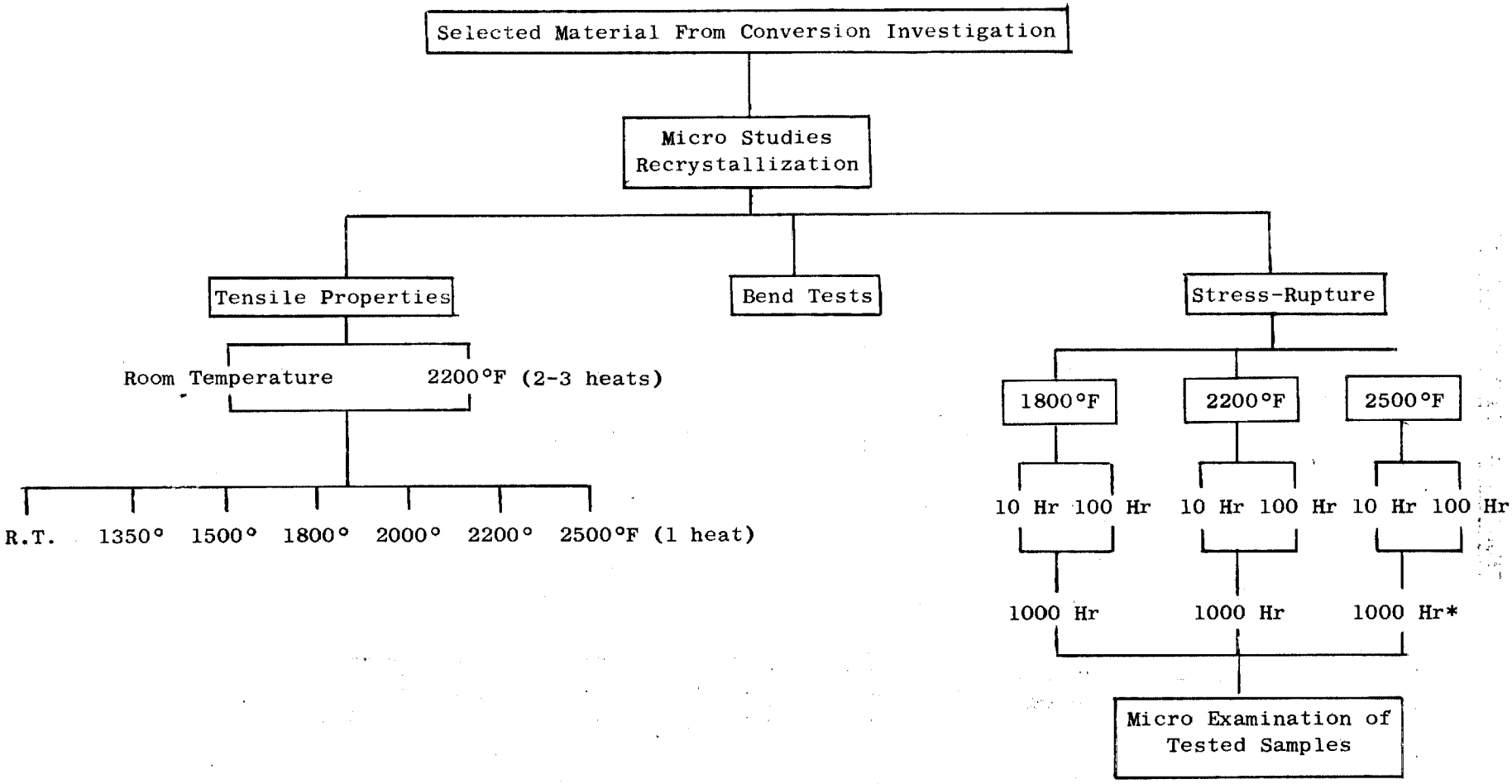


Figure 3

MECHANICAL PROPERTY EVALUATION



*Subject to Testing Equipment Limitations

2. Weld Ductility - To prepare weld samples, 1/2" x 2" blanks of AS-55 sheet will be placed in an evacuated and helium purged welding tank. An arc will be passed along the mid-line of the longitudinal axis of each blank, producing a "welded" structure (100% penetration). Bend tests will be used to determine weld ductility in the as-welded condition and after various thermal treatments. Several room temperature and hot tensile tests will be made to define these factors more completely. The time/temperature conditions likely to result in weld embrittlement will be examined as well as the effectiveness of post-weld heat treatments for maintaining weld ductility at the critical time/temperature conditions (Figure 4).

3. Corrosion Behavior - Selected AS-55 material will be used to fabricate tab specimens and capsules by rolling and welding. Duplicate tab specimens will be enclosed inside of the capsule, and another specimen will be attached to the outside of the capsule as a control sample. Identical Cb-1Zr alloy capsules and tab specimens will be prepared and tested simultaneously with the AS-55 capsules. Procedures for loading the capsule with potassium will be similar to those currently in use at FPLD, as illustrated in Table III and Figure 5. It is planned to conduct refluxing capsule tests in the 2000°F temperature range for periods up to 1,000 hours in a vacuum. Following the exposures, the materials will receive a detailed metallurgical evaluation.

Figure 4

EVALUATION OF WELD DUCTILITY

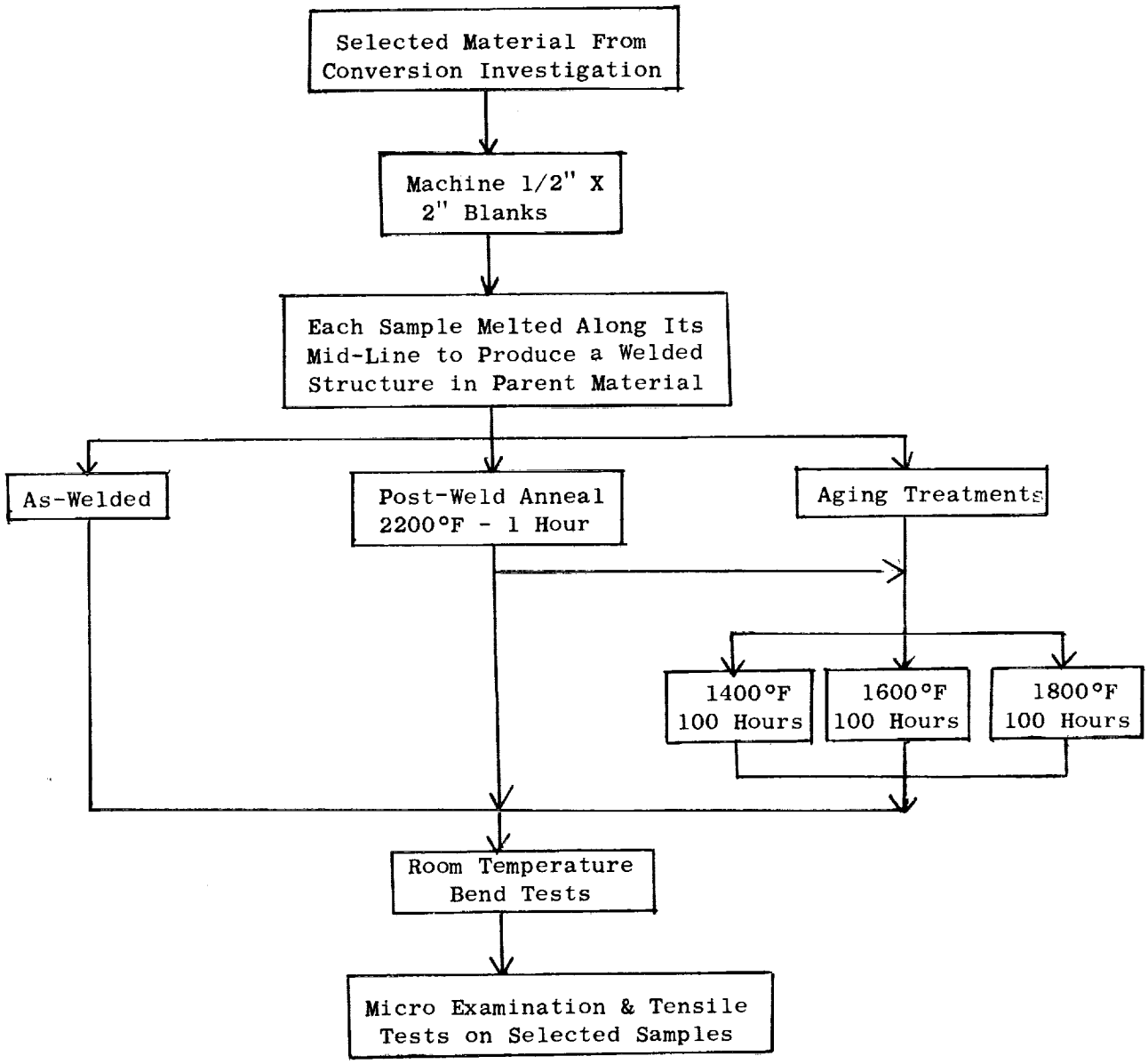


Table III

PROCEDURE FOR FILLING CAPSULES WITH POTASSIUM

(REFER TO FIGURE 5)

1. Evacuate the capsule loading system to below 1 x 10^{-3} torr.
2. Heat the system to above 150°F to permit outgassing.
3. Close the vacuum valve (A) to seal the capsule under a vacuum, and close vacuum valve (B) to isolate the system from the vacuum pumps.
4. Flush the system, other than the capsule, with argon by opening valves (C and D).
5. Re-evacuate the system to below 1 x 10^{-3} torr by closing valves (C and D) and opening valve (B).
6. Re-fill the system with argon.
7. Close one argon valve (D) and leave the other argon curtain flow valve (E) open.
8. Force potassium through the system by opening valve (F) and the potassium storage container valve (G) until potassium flows out of the argon curtain opening.
9. Allow the potassium to stay in the system for 3 to 5 minutes.
10. Force new potassium into the system as in 8.
11. Close the valve (C) to seal potassium in a sampling tube, and close valve (F) to confine a measured amount of potassium.
12. Open the capsule valve (A) and simultaneously open the argon valve (D) to force the measured length of potassium into the capsule.
13. Seal the capsule by closing valve (A), pinching down the tubing, and welding in a vacuum by electron beam.

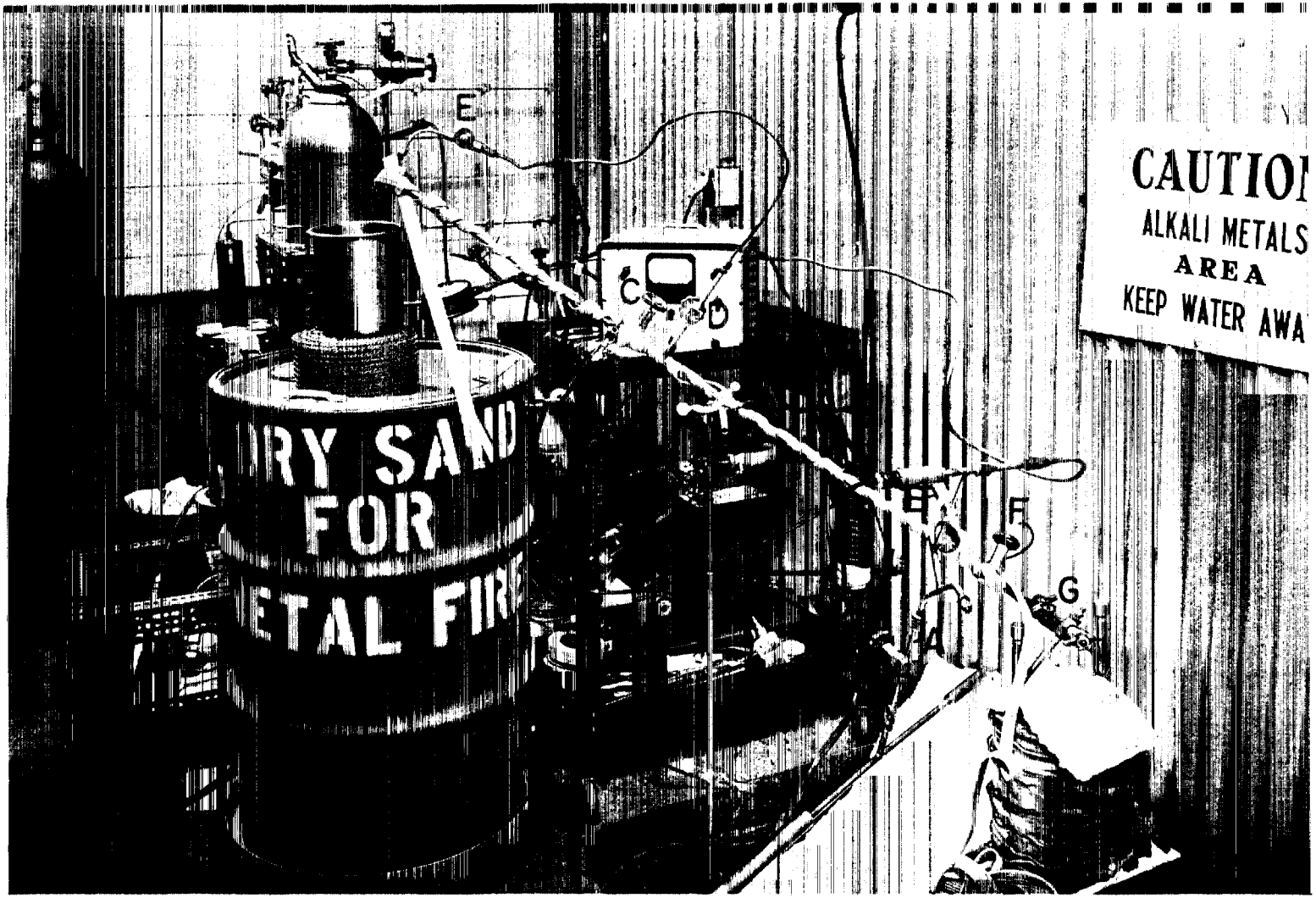


Figure 5. Apparatus for Filling Capsules with Potassium.

IV. PROGRAM STATUS

A. Materials Procurement

1. Electrode Material - Procurement of all raw materials for the electrodes is complete. The columbium metal for the major portion of the program was obtained from Kennametal, Inc. as -35 mesh powder. Smaller quantities of higher purity columbium in the form of spheres was obtained from the Du Pont Co. Tungsten was obtained from the General Electric Co., and zirconium hydride was obtained from Metal Hydrides, Inc.

   Because yttrium hydride was not readily available, it was produced at FPLD from solid yttrium metal. The yttrium hydride was made by heating the metal at 1900°F for 24 hours in a double retort hydrogen atmosphere furnace (dew point of -110°F) followed by a slow cool under flowing hydrogen. The resulting hydride, with an oxygen content of 1.92%, was quite friable, and after a slight amount of crushing, the particle size was below -20 mesh.

   A listing of the materials procured for this portion of the program is shown in Table I. A more detailed chemical and sieve analysis for the Kennametal columbium powder is presented in Table IV.

Table IV

CHEMICAL AND SIEVE ANALYSIS OF KENNAMETAL COLUMBIUM POWDER

Lot No. Bl. 287
Weight 144 Pounds

Chemical Analysis		Sieve Analysis	
Element	Weight %	Mesh Size	%
Carbon	0.06	-35+60	49.2
Oxygen	0.06	-60+100	17.8
Nitrogen	0.03	-100+150	9.3
Tantalum	0.08	-150+200	8.6
Iron	0.01	-200+325	5.7
Silicon	0.01	-325	9.4
Titanium	0.01		

Table V

WEIGHTS OF ALLOYING ADDITIONS

	Electrodes (1-19)		Electrodes (20,21)		Electrodes (22,23)	
	Nominal %	Weight (Kg)	Nominal %	Weight (Kg)	Nominal %	Weight (Kg)
Columbium	93.0	2.6040	93.8	2.6264	93.0	6.0915
Tungsten	5.0	0.1400	5.0	0.1400	5.0	0.3275
Zirconium (Hydride)	1.0	0.0280	1.0	0.0280	1.0	0.0655
Yttrium (Hydride)	1.0	0.0280	0.2	0.0056	1.0	0.0655

2. Jacketing Materials - The following items have been received to provide jacketing material for the initial high temperature breakdown of the AS-55 billets:

Frame Material - 1" x 1" Ti-75A bar stock - 25 feet.

Cover Plates - 0.060" 6Al-4V-Ti sheet - 10 sq. feet.

3. Cb-1Zr Alloy - Cb-1Zr alloy sheet, 4" wide x 0.100" thick x 10 feet long (1-foot multiples), has been received from the Haynes Stellite Co. The designated compositional requirements for the interstitial elements were:

0.030 O_2 max.

0.015 C max.

0.030 N_2 max.

This sheet is being inspected prior to processing into capsules for comparative corrosion tests.

B. Electrode Section Preparation

Twenty-three AS-55 alloy electrode sections were prepared at Kennametal, Inc. Twenty-one sections were made using the -35 mesh columbium powder supplied by Kennametal, and two were made using the Du Pont columbium spheres. A 1.0% yttrium addition (hydride) was made to the nineteen of the twenty-one sections containing columbium powder and to the two sections containing columbium spheres. Two electrode sections containing columbium powder were made with only 0.2% yttrium additions. The yttrium additions and the other alloying

additions, including 5% tungsten and 1% zirconium (as hydride), were weighed out separately at FPLD and added to the columbium powders at Kennametal, Inc. Weights of each element used in preparing these charges are shown in Table V.

Each mixture of Kennametal columbium powder and alloying additions was blended in a twin-shell blender for 15 minutes. In order to facilitate further blending of the material, the mix was turned end-to-end in a polyethylene bag prior to "slugging" in a 2-inch diameter chamber. In the charging of the chamber, the polyethylene bag was inverted over the mouth of the chamber, and the blend was lowered into position.

A trial electrode section was slugged and hydrostatically pressed at 33,000 psi to determine the amount of bowing as well as the section size. This pilot electrode was determined to have a 3/16-inch bow, a diameter of 1.78 inches, and a length of 9.5 inches.

The next seventeen powder mixtures with 1% yttrium additions were slugged without jogging, bundled together, and hydrostatically pressed using explosive techniques. In the explosive pressing operation, the slugged compacts were placed into a 16-inch gun-barrel chamber along with a sufficient explosive charge. The breach was sealed and the charge ignited to produce an isostatic pressing environment of 31,000 psi. The weights and dimensions of these sections are recorded in Table VI.

Table VI

WEIGHTS AND DIMENSIONS OF ELECTRODE SECTIONS

Section	Weight (grams)	Avg. Diameter (in.)	Length (in.)	Remarks
1	2550	1.78	9.00	1.0% Yttrium Addition
2	2805	1.77	9.50	" " "
3	2805	1.76	10.50	" " "
4	2805	1.79	10.00	" " "
5	2800	1.82	9.50	" " "
6	2805	1.79	10.00	" " "
7	2805	1.81	10.00	" " "
8	2805	1.78	10.00	" " "
9	2805	1.79	10.00	" " "
10	2805	1.81	10.00	" " "
11	2805	1.81	10.00	" " "
12	2805	1.78	9.75	" " "
13	2800	1.78	10.00	" " "
14	2805	1.78	10.00	" " "
15	2805	1.79	10.00	" " "
16	2805	1.78	10.00	" " "
17	2805	1.77	10.50	" " "
18	2805	1.77	10.35	" " "
19	2530	1.79	9.75	
20	2800	1.77	9.75	0.2% Yttrium Addition
21	2800	1.74	10.00	" " "
22	1350	1.5 X 1.5*	5.00	Low Interstitial Cb
23a	3200	1.5 X 1.5*	12.00	" " "
23b	1980	1.5 X 1.5*	7.50	" " "

*Rectangular cross section

All eighteen electrode sections were wrapped in tantalum foil and vacuum sintered as described in Table VII. The furnace was cooled below 400°F under vacuum before discharging.

Materials for the four remaining modified AS-55 electrode sections and for the nineteenth typical section of AS-55 were weighed and shipped to Kennametal, Inc. Three electrode sections, two containing the lower yttrium content (0.2%) and one typical AS-55 section, were explosively pressed in the same manner as described above. Mixes for the other two modified AS-55 electrode sections containing the high purity columbium spheres were mechanically pressed at 60,000 psi in a 1-1/2-inch x 25-inch die. The mix with the columbium spheres was divided into five separate groups. Each of these groups was blended by dumping back and forth in a polyethylene bag. The die cavity, divided into five equal segments by metal dividers, was loaded with each of the five charges. Three of the dividers were removed, resulting in a 20-inch long and a 5-inch long electrode. This was done to accommodate the sintering furnace which has a 20-inch maximum hot zone. The large rectangular electrode section was quite weak, and in wrapping with tantalum foil, the section broke at one of the regions where a divider had been located previously. This electrode section has been repaired by welding. The weights and dimensions of these electrodes are recorded in Table VI.

Table VII

VACUUM SINTERING RECORD FOR ELECTRODE SECTIONS 1 TO 18

Furnace Run No. 266-7

Time	Temperature (°F)	Power (KW)	Vacuum (Microns)
7:30 A.A.	Room	4	0.3
8:30 A.M.	Dull Red	6	4.0
9:30 A.M.	1800	8	160.0
10:30 A.M.	2450	10	40.0
11:05 A.M.	2800	6	6.0
11:20 A.M.	2800	Off	4.5

Table VIII

VACUUM SINTERING RECORD OF ELECTRODE SECTIONS 19 to 23

Furnace Run No. 273-7

Time	Temperature (°F)	Power (KW)	Vacuum (Microns)
10:00 A.M.	Room	10	0.3
11:00 A.M.	1450	8	50.0
12:00 P.M.	2150	6	5.5
1:00 P.M.	2370	20	3.8
1:20 P.M.	2800	8	2.0
1:35 P.M.	2800	Off	1.5

After wrapping the remining pressed electrode sections in tantalum, they were vacuum sintered as indicated in Table VIII. Again, the furnace was cooled below 400°F under a vacuum before the electrodes were removed. It was observed that the rectangular electrodes had many longitudinal as well as transverse cracks which are believed not to be detrimental with respect to melting.

C. Arc Melting

Eight ingots have been arc melted in the facilities of the General Electric Company Research Laboratory. Figure 6 is a view of the overall arc melting facilities, and a schematic diagram of the arc melting furnace is shown in Figure 7.

All arc melting was performed using direct current with the electrode negative. The D.C. rectifiers which were used to supply the power are so regulated as to maintain a constant current at a predetermined level. The desired melting voltage was obtained by controlling the arc gap by means of an electrode drive motor which obtains a signal through a thymotrol power supply from the relative voltage between a preset reference and the actual arc melting voltage. This electrode drive mechanism cannot retract the electrode automatically. The lack of electrode "hunting" in these and other experiments is attributed to the unidirectional action of this electrode feeding device. Eleven thousand amperes at 30 volts were available for melting.

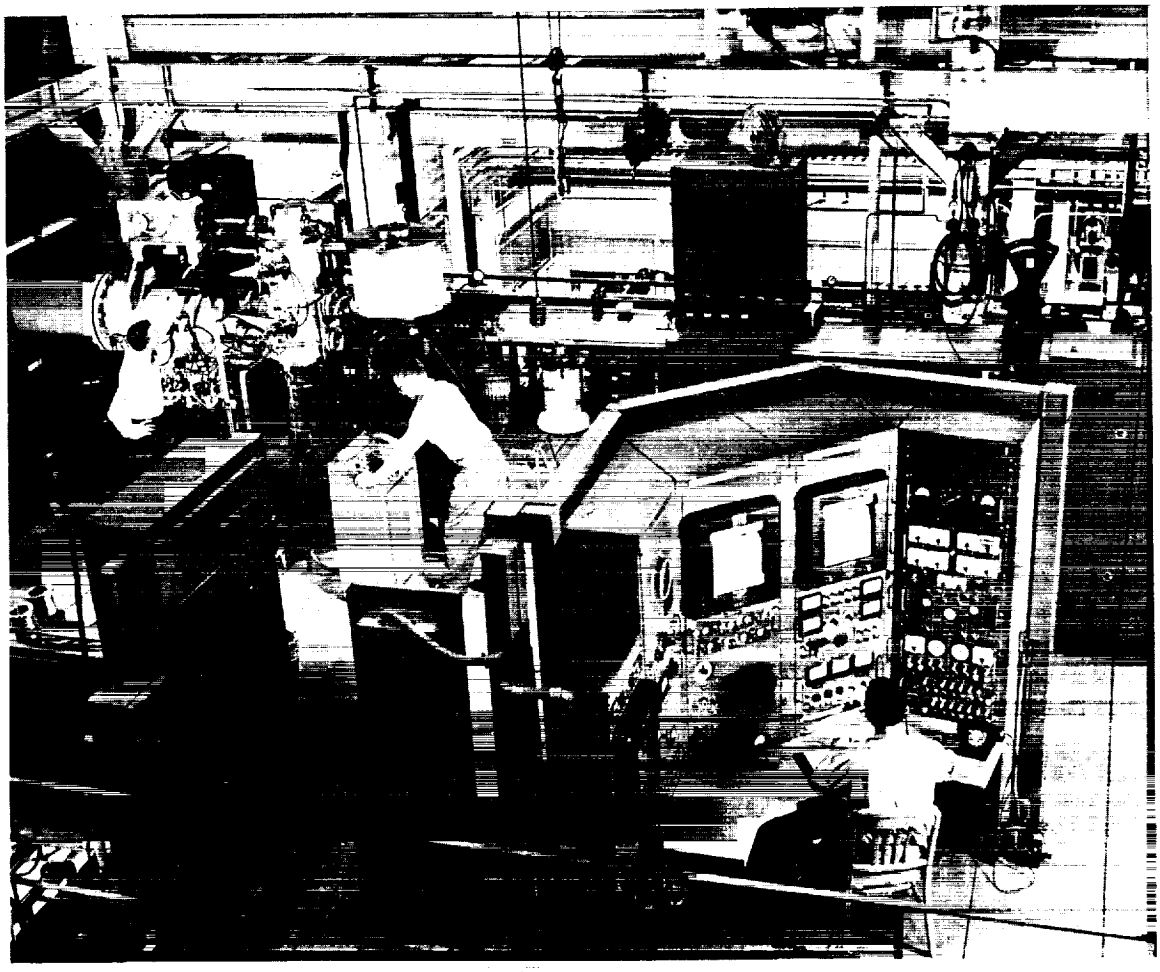
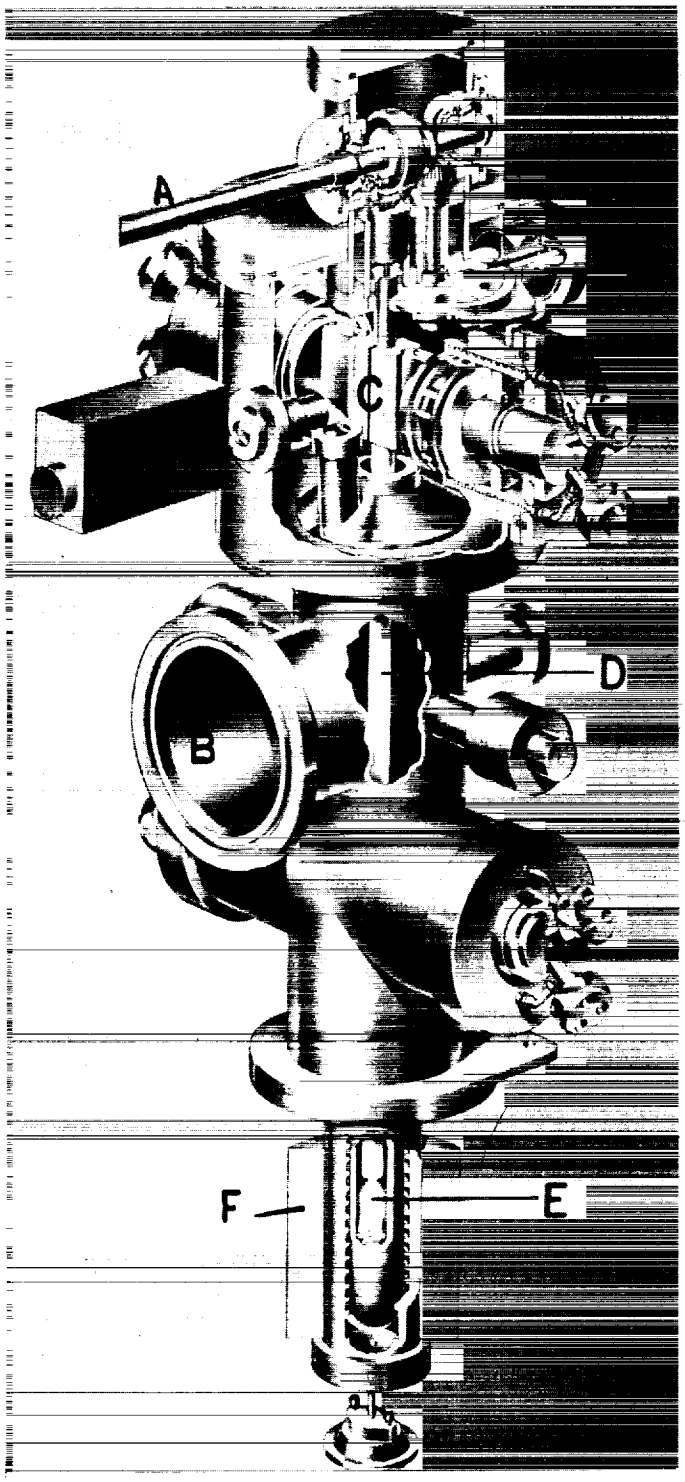


Figure 6. Arc Melting Facilities - General Electric Co. Research Laboratory



A - Electrode Drive

B - Vacuum Port

C - Electrode "Shoe" Contact

D - Electrode

E - Arc

F - Stirring Coil

Figure 7. Schematic Diagram of the Arc Melting Furnace.

A 16-inch oil-diffusion pump which was backed up by an oil ejector pump and an oil-sealed mechanical pump comprised the vacuum system This system is capable of pumping at a speed of 4300 ℓ/sec at a furnace pressure of 1×10^{-3} torr. Melting pressures were continuously read at two places in the system. The first was the pressure in the 16-inch furnace manifold.

The second pressure was measured adjacent to the top of the crucible. This pressure was used as a first approximation of the pressure which existed in the purification zone of the crucible. It is this pressure which will be referred to as the melting pressure in this investigation.

The cooling of the copper crucible was accomplished by using a spiral-grooved water jacket. Cooling water at 10° to 20°C, flowing at approximately 30 gallons per minute, was used. No difficulties have been encountered using as high as 27 kw of melting power.

The amount of molten pool stirring was controlled by a standard direct current stirring coil. The field strength in oersteds has been measured at the center of the empty coil, and the value read on the control panel, in amperes, can be converted directly to the field strength value.

The first ingot (12 pounds), NAS-551, was melted in a 3-inch diameter crucible. Melting conditions were varied to determine the approximate midpoint for melting the remaining electrodes. It was determined that good control of melting was obtained with an average voltage of 28.4 volts, with a current of 5,750 amperes, and with a stirring reading of 0.2 amperes (approximately 20 oersteds). Arcing to the copper crucible wall and arc extinction occurred half way through the melt as a result of the deliberate decrease in power to establish midpoint conditions. This arc-over resulted in the sticking of the ingot to the crucible. After restriking the arc and completing the melt, the ingot was removed by drilling a hole in the bottom of the crucible, inserting a ram, and applying pressure.

Midpoint melting conditions were selected as follows:

Voltage - 28 volts

Current - 5,750 amperes

Stirring - 0.2 amperes ($\sim$20 oersteds)

E/M Ratio - 0.34

The second ingot (6 pounds), NAS-552, was melted at the midpoint conditions, except that a larger diameter crucible, 3-5/8 inch, was used to obtain an electrode-to-mold area ratio of about 0.23.

The third ingot (12 pounds), NAS-553, was melted at the midpoint conditions.

The fourth ingot (6 pounds), NAS-554, was melted in a 2-1/2-inch diameter crucible to obtain an electrode-to-mold ratio of approximately 0.49. On the initial trial to melt this ingot, the electrode sections fractured at the sintered joint. By increasing the sintering time for joining the sections, the second attempt to melt was successful.

The fifth ingot (12 pounds), NAS-555, was melted, using the amount of stirring as a variable. In the lower half of the ingot, the stirring field was held at 0.05 amperes ($\sim$ 4 oersteds), and in the upper half, the stirring was increased to 0.35 amperes ($\sim$ 36 oersteds). This 12-pound ingot accounts for two of the intended 6-pound ingots discussed in the technical plans and illustrated in Figure 1.

The sixth ingot (6 pounds), NAS-556, was melted using a lower voltage than the midpoint, approximately 34 volts.

The seventh and eighth ingots (6 pounds each), NAS-557 and NAS-558, were melted with the current as a variable, 5,250 and 6,250 amperes, respectively.

The first five ingots have been sawed longitudinally into two halves, and one half from each ingot was macroetched as shown in Figures 8, 9, and 10. It is immediately apparent that these ingots have a fine-grained structure, in comparison to the normally coarse, columnar grains found in arc melted unalloyed columbium. Evidence of hot topping can be observed in the macrostructure of all the ingots.

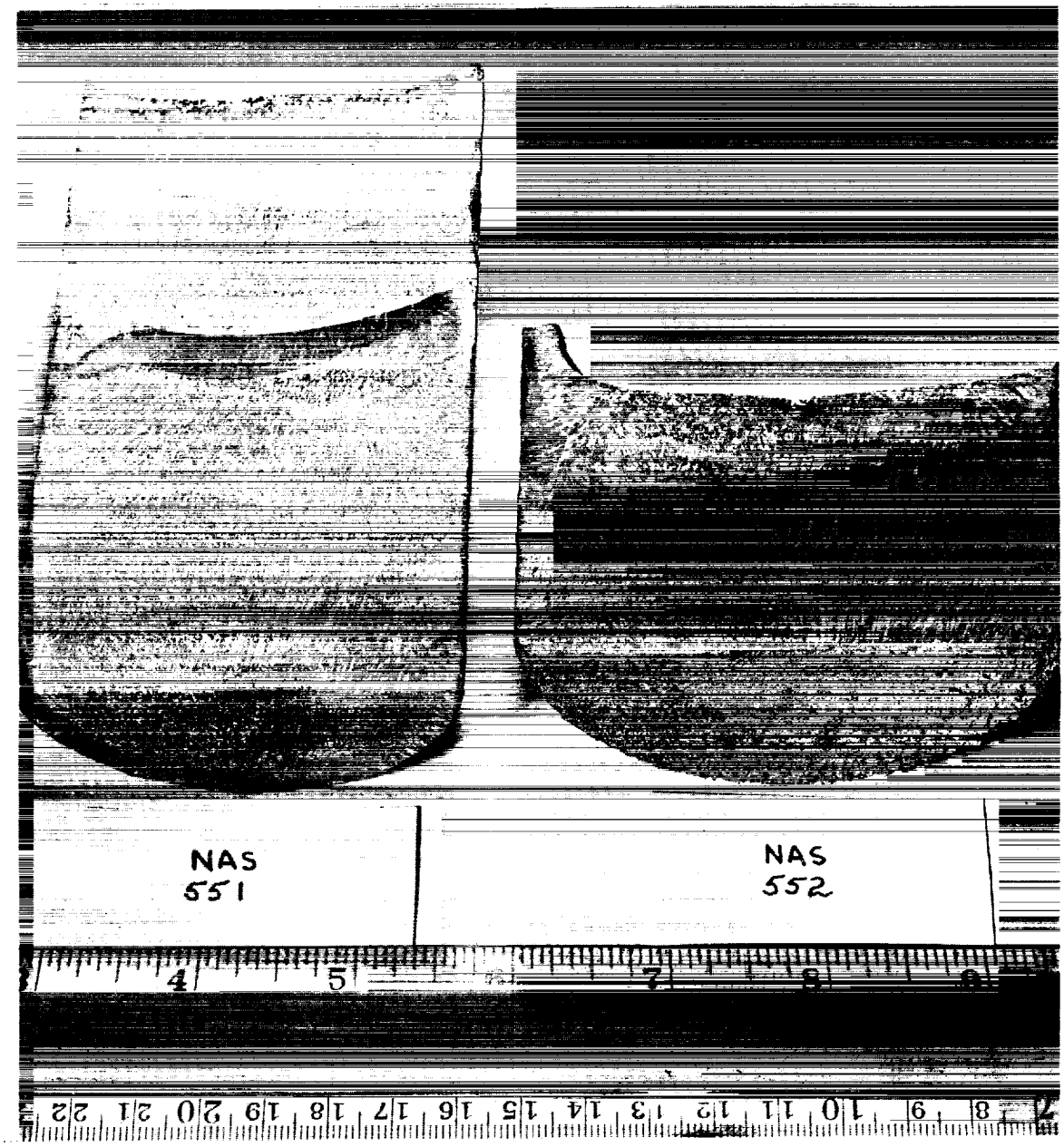


Figure 8. Macrostructure of Ingots NAS-551 (Pilot Ingot) and NAS-552. The Location in NAS-551 where the Arc was Extinguished by a Deliberate Reduction of the Current below 2,000 amperes is Evident.

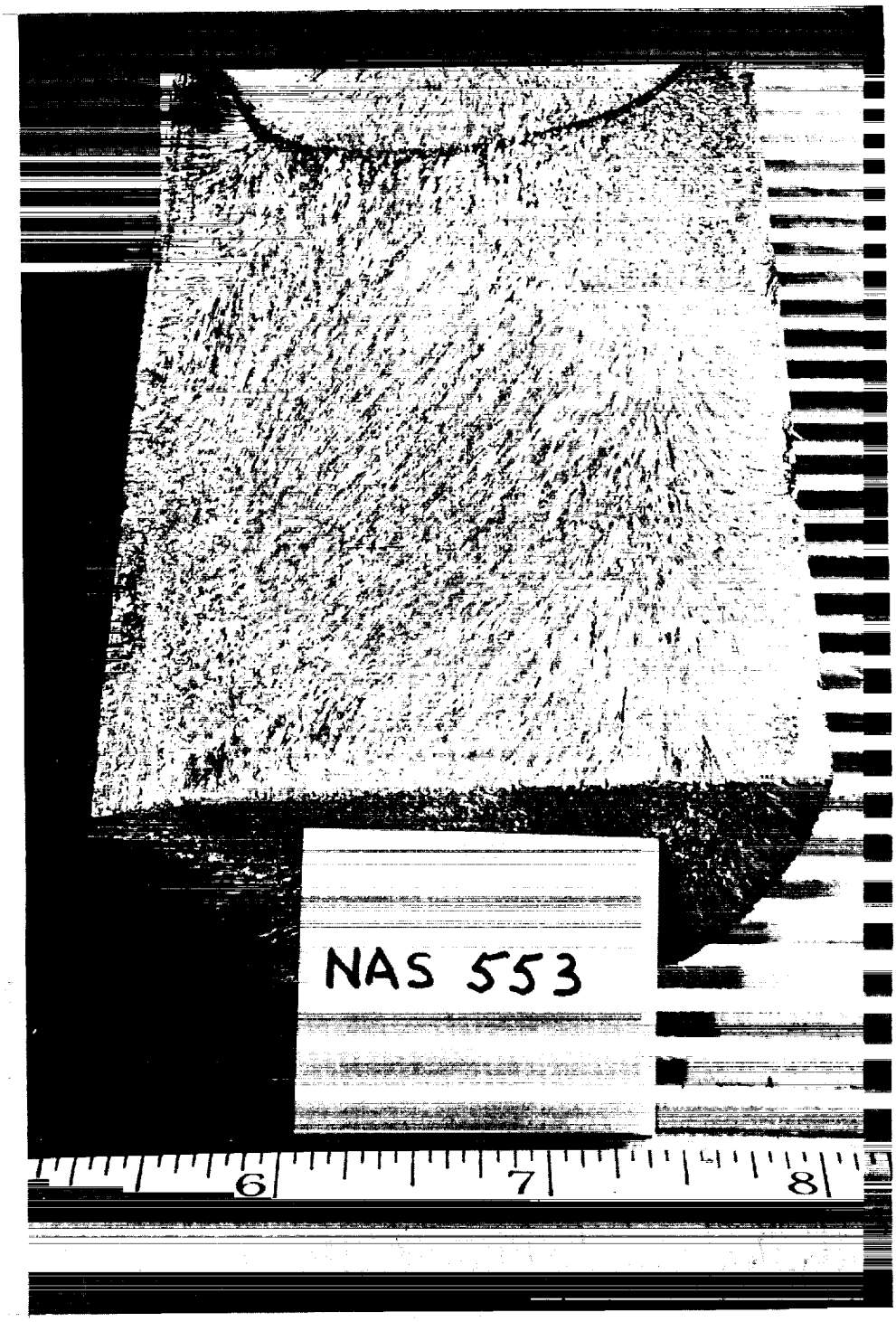
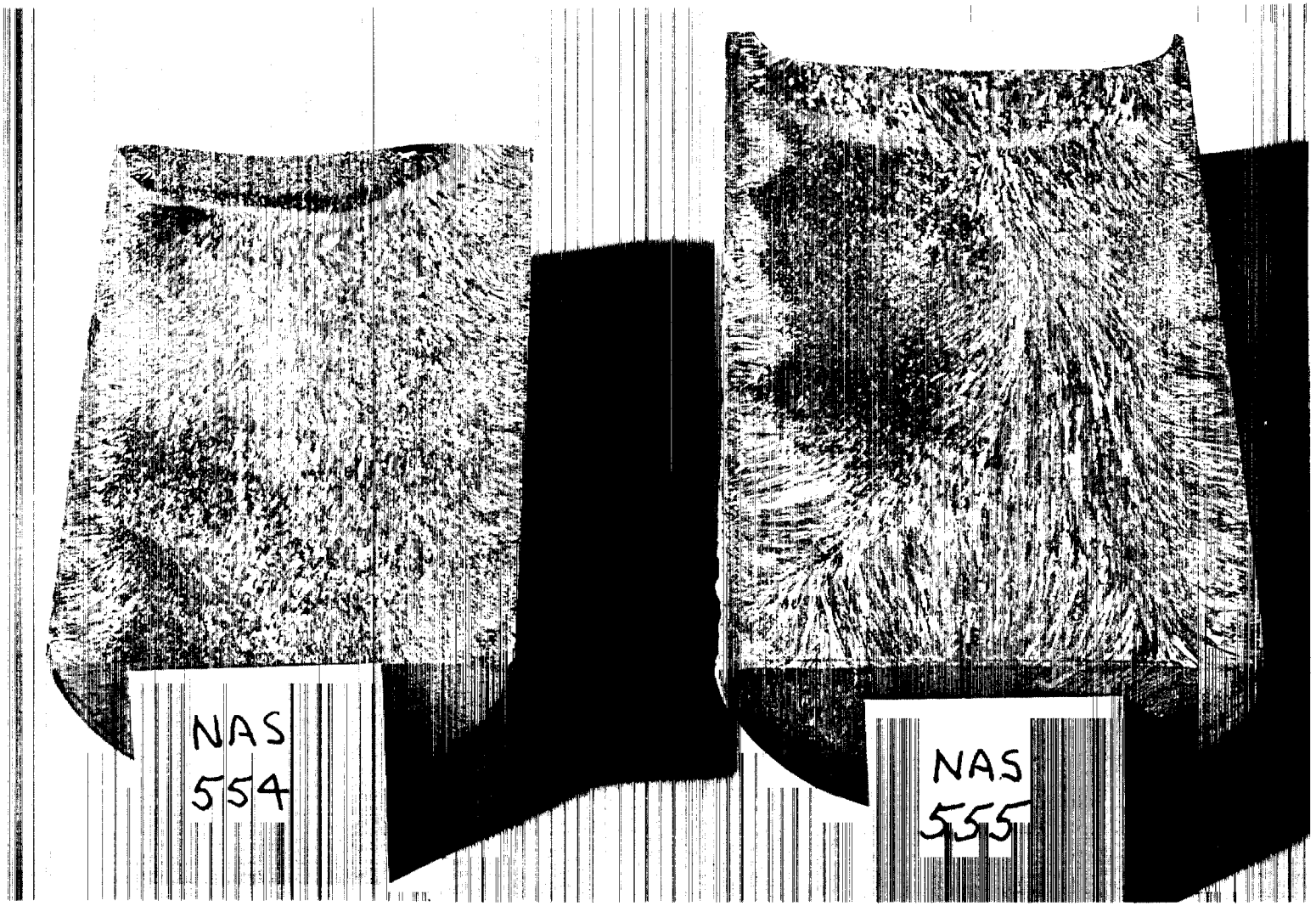


Figure 9. Macrostructure of Ingot NAS-553. All Melting Variables were at the Selected Midpoint.



10. Macrostructure of Ingots NAS-554 and NAS-555. In Ingot NAS-554 the Electrode/Mold Area Ratio was Increased from 0.34 to 0.49. The Effect of Increasing the Stirring from 4 to 36 oersteds during the Melting of NAS-555 can be Observed in the Macrograph.

An estimate of the grain size of NAS-551 is ASTM 2-3 in a transverse section and ASTM 1-3 in a longitudinal section. At approximately two-thirds of the distance from the bottom on the NAS-551 ingot, a discontinuity in the structure can be noted (Figure 8). This discontinuity occurred during melting when the current was deliberately decreased to below 2,000 amperes and the arc wandered to the copper crucible, as described previously.

In Figure 9, the cross section of ingot NAS-553 is shown. The grain size is estimated to be ASTM 1-4 in a transverse section and ASTM 1-3 in a longitudinal section.

The smaller 2-1/2-inch idameter ingot (NAS-554) is shown in Figure 10, along with ingot NAS-555 in which the stirring was varied. A marked effect of stirring can be noted in the NAS-555 ingot. The lower portion, with the stirring at approximately 4 oersteds, has the larger grain structure, and the upper portion, with the stirring at approximately 36 oersteds, has a much finer grain structure.

The weights and dimensions of five ofthe ingots are listed in Table IX.

Evaluation of the ingots will continue during the next quarter.

Table IX

WEIGHTS AND DIMENSIONS OF INGOTS

Heat No.	Length, in.	Diameter, in.	Weight, lbs.
NAS-551	5.75*	2.9	13.25
NAS-552	2.10*	3.6	7.68
NAS-553	4.92**	2.9	10.37
NAS-554	3.62*	2.4	6.37
NAS-555	4.45*	2.9	10.75

*Does not include skull
**Both ends cropped prior to delivery

Table X

WEIGHTS AND DIMENSIONS OF BILLETS

Heat No.	Length, in.	Width, in.	Thickness, in.	Weight, lbs.
NAS-551	5.20	2.05	1.05	3.50
NAS-553	4.87	1.90	1.12	3.25
NAS-554	3.40	1.60	0.92	1.56
NAS-555	4.30	1.75	1.22	2.87

D. Ingot Conversion

One half of the longitudinally sectioned ingots, NAS-551, 553, 554, and 555, were machined into billets (Figure 11) having the dimensions shown in Table X. Typical billets machined from the ingot halves of NAS-554 and 555 are shown in Figure 12.

Titanium frames and titanium alloy face plates were cut to size and tack welded in place around billets NAS-551 and 553. All edges, but one, were arc welded in a vacuum purged (10^{-4} torr), helium atmosphere welding chamber that had been gettered by puddling a titanium strip. When the welded packs were cooled, they were then placed in a 30 kv electron beam welding facility. After evacuation to less than 10^{-4} torr, the final edge was sealed. A completed pack (NAS-551) is shown in Figure 13.

Initial breakdown of billets NAS-551 and 553 was accomplished at the General Electric Company Research Laboratory. Both billets were soaked for 45 minutes at 2200°F in an argon atmosphere prior to processing. Billet NAS-551 was given one full blow with a 2,500-pound forging hammer which resulted in an overall reduction of 17%. After re-heating, three additional blows of the hammer imparted a reduction of 27%, or 40% total reduction of the pack. At this stage, the billet appeared quite sound and was returned to the furnace for re-heating. Further working was carried out on a two-high 24-inch Birdsboro rolling mill at a temperature of 2200°F. The pack was given two passes, without re-heat, to a final thickness of 0.305 inch, which reduced the thickness another 57%, or a total reduction, by forging and roll-

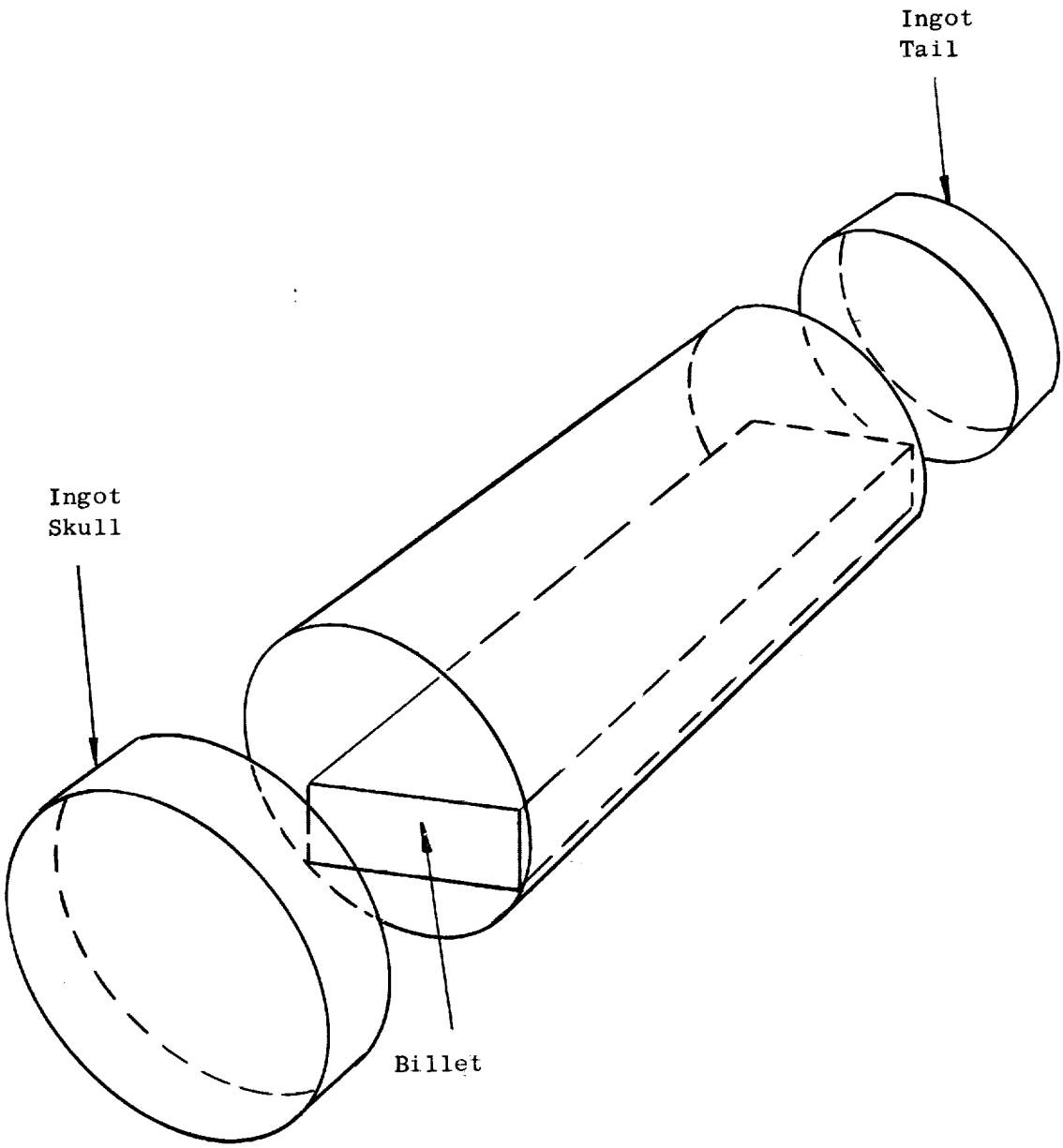


Figure 11. Schematic Diagram of an Arc Cast Ingot Sectioned to Obtain a Billet for Conversion to Sheet.

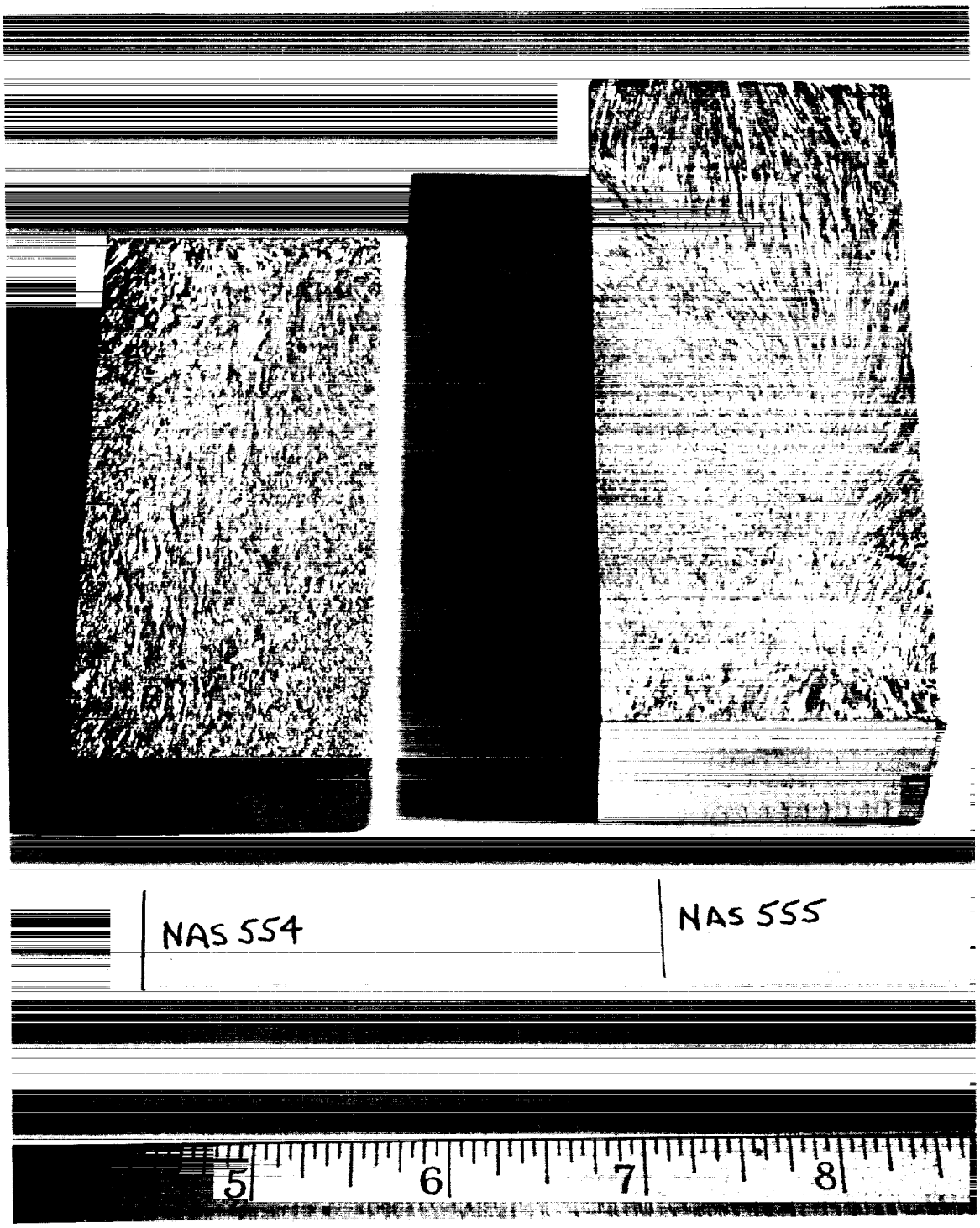


Figure 12. Billets Machined from Ingots NAS-554 and NAS-555.

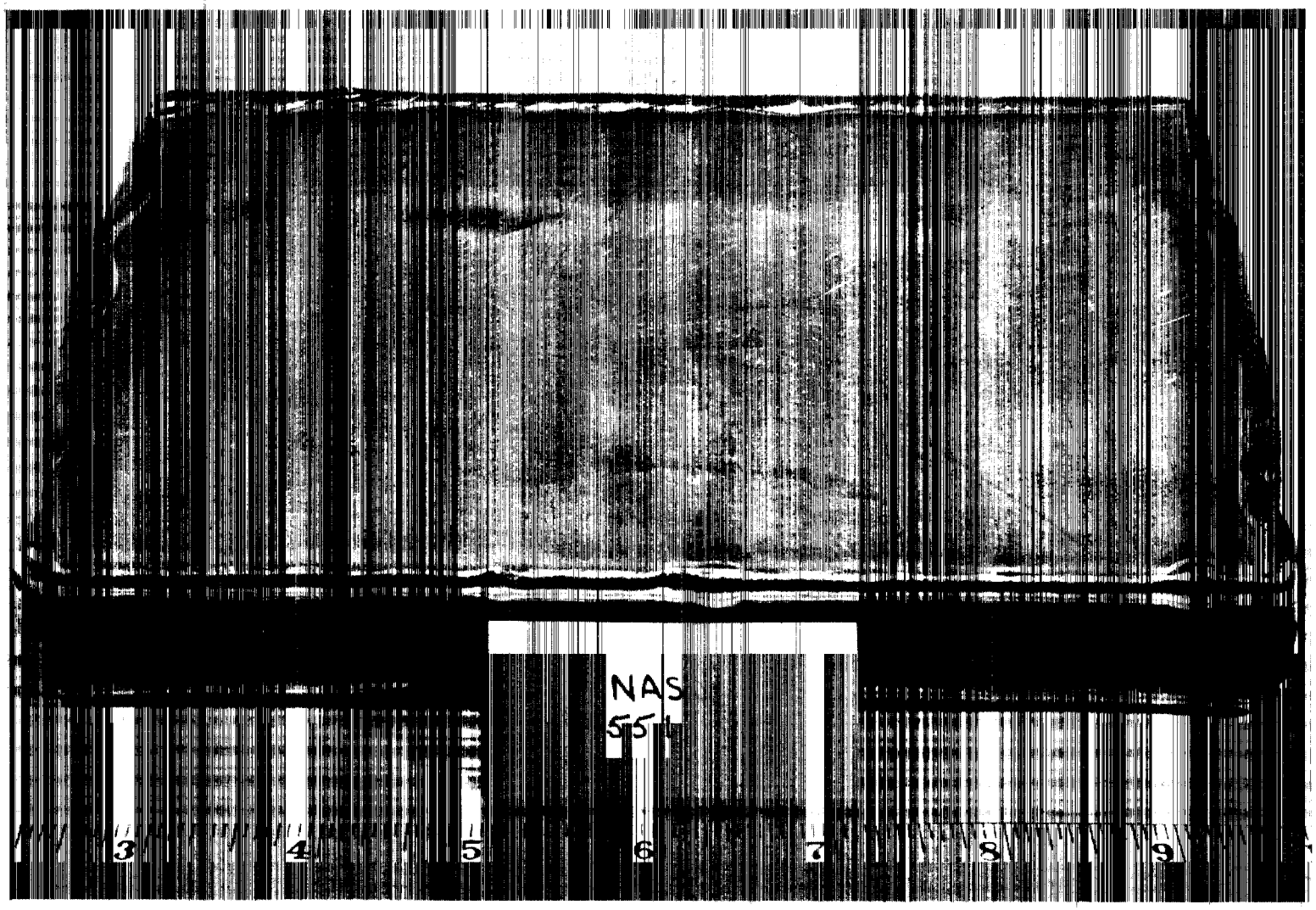


Figure 13. Titanium Clad Billet from Ingot NAS-551.

ing, of 74%. The rolling direction was in the long axis of the original ingot blank (Figure 14).

Billet NAS-553 was given four blows with the forging hammer at 2200°F without re-heating. At this point, a break in the cladding was observed at the interface of the titanium frame and the AS-55 core. This failure in the cladding can possibly be attributed a the time delay in forging, permitting the pack to cool to a temperature where the cladding/frame material at the perimeter of the core moved more readily than the cladding bonded to the AS-55 material and, thereby, causing separation at the interface. The work imparted to the billet by this one operation (four blows) resulted in a 43% reduction to a thickness to 0.707 inch.

The titanium frame was sawed from the NAS-551 plate, and the cladding was removed by pickling in a 10 HF - 30 HNO_3 - 60 H_2O solution. The exposed plate is shown in Figure 15. Note that the hot top and the area where the arc was re-struck in the original ingot are clearly discernible. The plate was sectioned for cold rolling according to the layout shown in Figure 16.

The middle or scrap section where the arc was extinguished and re-struck was rolled first to establish the proper amount of cold reduction per pass. A reduction of 20% in one pass at room temperature caused moderate to severe cracking along the mid-line of the entering edge of the plate. The piece was

Figure 14. Plate from Ingot NAS-551 with the Titanium Cladding after Forging 40% and Rolling 57% at 2000°F.

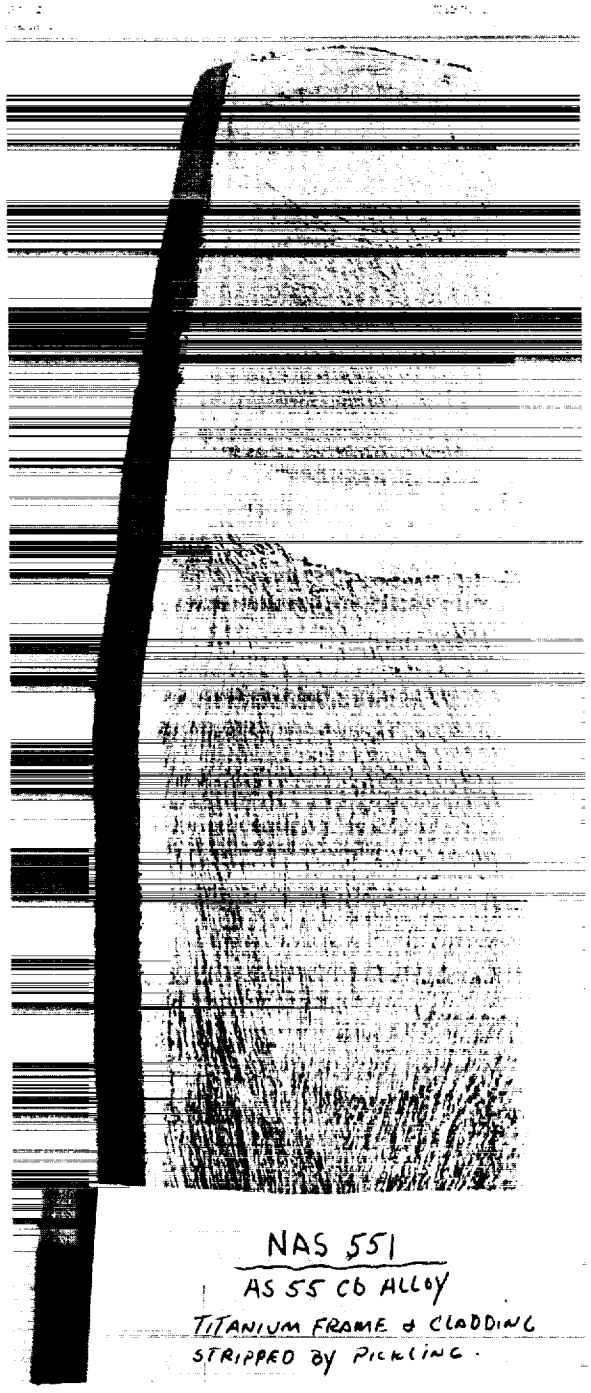


Figure 15. Plate from Ingot NAS-551 (0.240-inch Thick) after Removal of the Titanium Cladding by Pickling. The Hot Top Area and the Area where the Arc was Re-struck will be Removed prior to Evaluation of the Sheet.

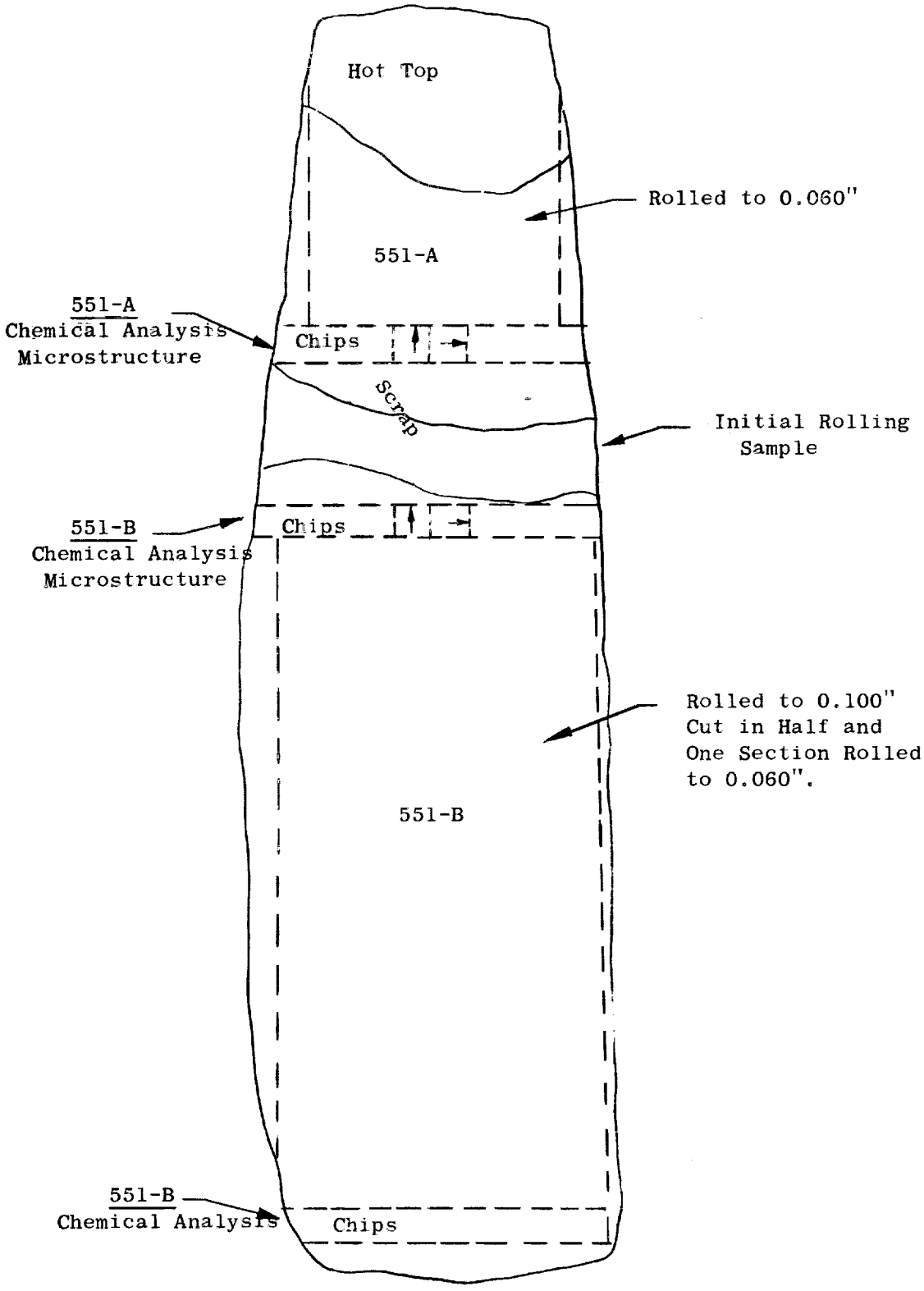


Figure 16. Disposition of the 0.240-inch Thick Plate from Ingot NAS-551.

turned end for end, and lighter passes reduced the severity of the cracking. Additional, rapid, light reductions showed no tendency to propagate the original cracks, and 0.060-inch thick material was produced with only minor edge defects appearing on the final few passes.

Plate NAS-551-A was cold rolled 75%, 10% per pass, to 0.060-inch thick sheet without difficulty. NAS-551-B was rolled to 0.100-inch thick sheet and cut in half. One half of this sheet was rolled to a 0.060-inch thickness. The other half will be given a vacuum anneal for 1 hour at 2200°F and cold rolled to 0.060-inch thick sheet. Observations will be made on the effect of this intermediate anneal on rollability, surface finish, and properties of the sheet. Figure 17 shows the 0.060-inch thick sheet after being cleaned by a pickling operation. A recapitulation of the work imparted to ingot NAS-551 is shown below.

Operation	Temperature, °F	% Reduction
Forging	2200	40
Rolling	2200	57
Rolling	R.T.	75

Using the original billet thickness (1.05 inches) and the final sheet thickness (0.060 inch), a total reduction of 94% was obtained without difficulty.

The decision was made to warm roll billet NAS-553 at 2200°F, assuming that heating in argon and rapid handling of the billet between

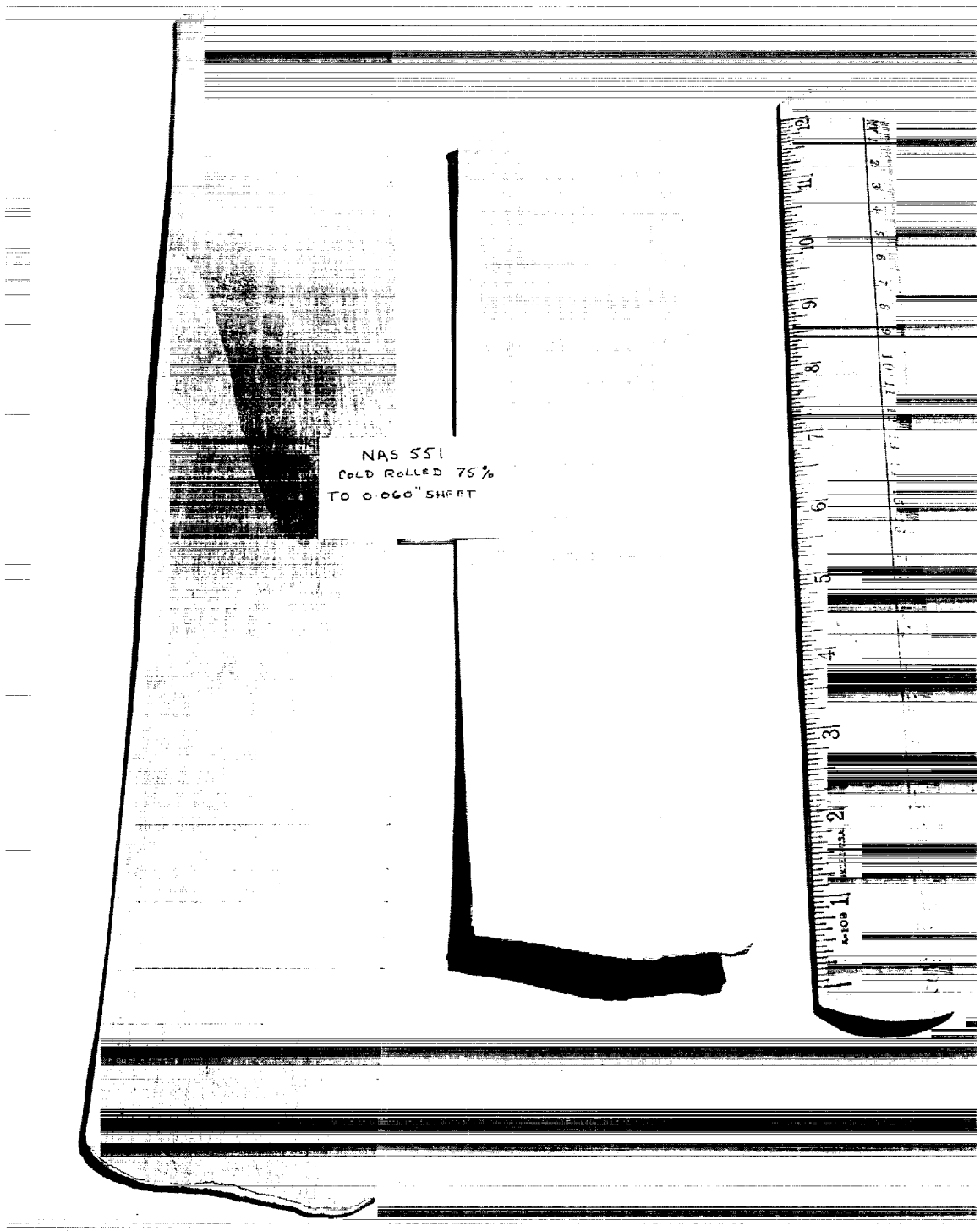


Figure 17. Cold Rolled (75%) 0.060-inch Thick sheet Produced from Ingot NAS-551. The Hot Top will be Removed Prior to Evaluation of the Sheet.

passes would minimize contamination through the small break in the cladding. Two passes transverse to the ingot axis were used to widen the plate, and three passes parallel to the ingot axis (cross rolled) were made, resulting in a reduction of 48%. The defective edge areas, due to oxidation through the fractured titanium, were cropped, and the cladding was removed by pickling. Further reduction of the resultant 0.320-inch thick plate will be accomplished at room temperature.

Additional sheet will be processed and evaluated during the next quarter.

E. Corrosion Testing

The reflux capsules to be fabricated from AS-55 and Cb-1Zr alloy sheet will be tested at temperatures near 2000°F in a high vacuum. The vacuum equipment to be used for this work consists of a 18-inch diameter x 30-inch high, bakeable chamber connected to a 400 ℓ/sec Vac-ion pump (Figure 18). When the chamber is cold and empty, a vacuum of 2×10^{-10} torr is obtained.

A single test assembly, incorporating a tantalum strip heater and six tantalum radiation shields, has been constructed and operated with a 1.05-inch O.D., 0.154-inch wall, 11-inch long Cb-1Zr alloy capsule filled with about 10 grams of slagged, filtered, distilled, and hot trapped potassium. The test was conducted as a preliminary examination of the heater and shielding design, power requirements, temperature distribution along the capsule, and vacuum capabilities of the system. Temperature was monitored with three,

Figure 18. Ultra-High Vacuum System (10^{-10} torr Range) to be Used for a Study of the Corrosion Behavior of AS-55 and Cb-1Zr in Potassium. The Chamber is 18 inches in Diameter and 30 inches High.

sheathed Ni/Ni-13Mo thermocouples placed at the top, middle, and bottom of the capsule. The assembly, mounted in the vacuum chamber, is shown in Figure 19.

This trial run was successfully conducted with the bottom wall temperature at 2000°F for 100 hours, using about one-kilowatt of power. The temperature of the upper portions of the capsule was between 1600° and 1700°F. As the test progressed, the system pressure decreased from initial values near 1×10^{-6} torr to 5×10^{-9} torr.

For the subsequent corrosion tests, a multi-capsule heater assembly will be constructed to expose four capsules simultaneously.

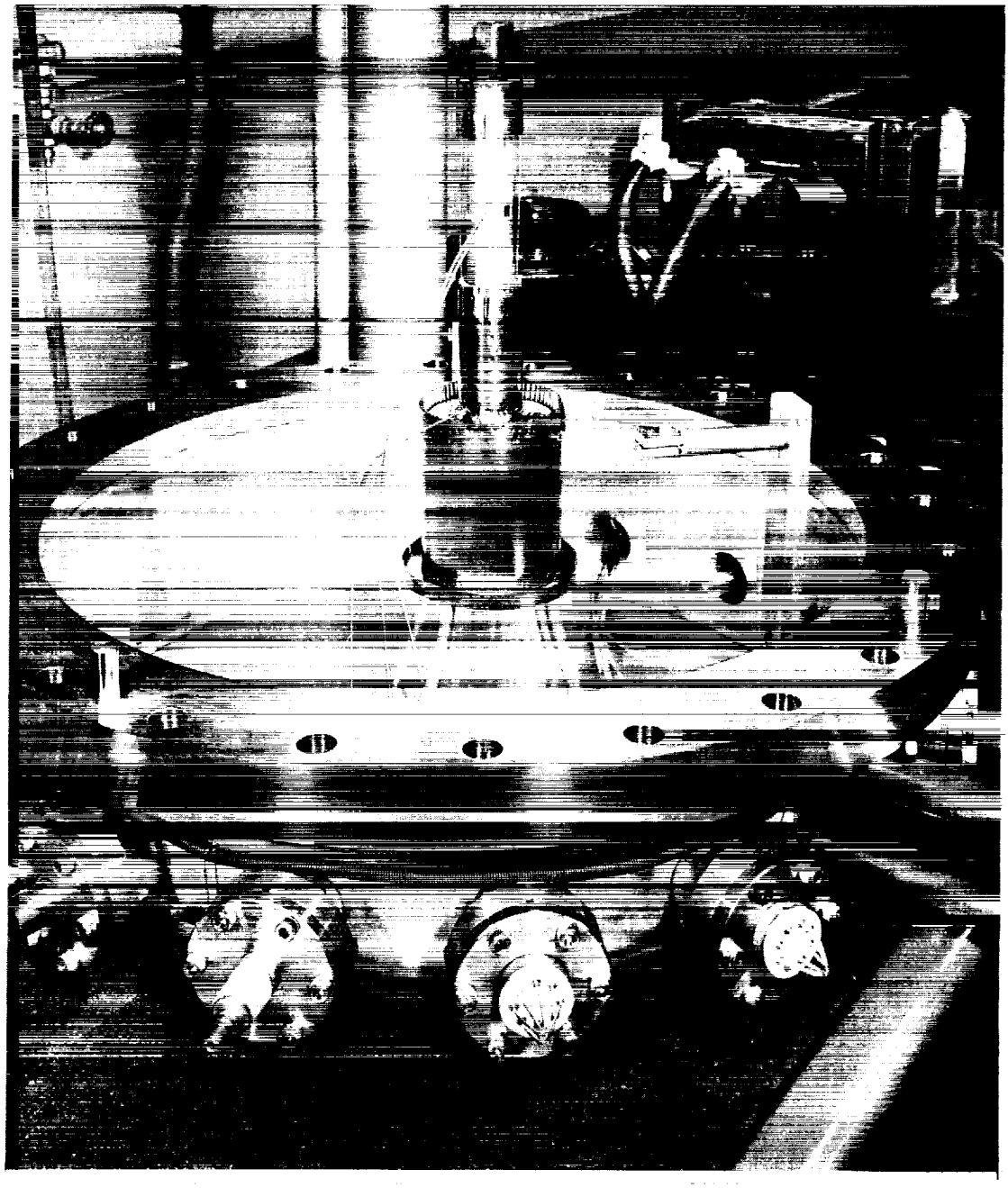


Figure 19. Single Reflux Capsule Heater Assembly Positioned in the Lower Section of the Ultra-High Vacuum Chamber.

V. FUTURE PLANS

The following work will be conducted during the next quarter:

1. Complete the melting and evaluation of the remaining ingots, including the microstructural examination.

2. Fabricate sufficient quantities of sheet to initiate the investigations of weld ductility and mechanical properties.

3. Complete evaluation of the reflux capsule heater design and initiate the corrosion tests. Expectations are that 1000-hour corrosion exposures will be completed close to the end of the quarter.

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